

Application Serial No: 10/509,463  
Responsive to the final Office Action mailed on: December 24, 2009

### **REMARKS**

This Response is in response to the Office Action mailed on December 24, 2009.  
Claims 5 and 7-11 are pending with claims 10 and 11 being withdrawn.

#### **§103 Rejections:**

Claims 5, 7 and 9 are rejected as being unpatentable over DeLozanne (US Patent No. 5,004,721) in view of Higuchi (US Patent No. 5,079,224) and further in view of Yanai (US Patent No. 4,511,594). This rejection is traversed.

Claim 5 is directed to an apparatus for manufacturing a thin film in which the thin film is formed on a supporting base that recites, among other features, an electron beam source that is arranged in the vacuum container and emits an accelerated electron beam to be used to evaporate the first thin film material by heating using an electron beam heating method. Claim 5 also recites that the electron beam evaporation source, the electron beam source and the resistance heating evaporation source are arranged so that a path along which the accelerated electron beam emitted from the electron beam source reaches the electron beam evaporation source intersects with a line segment connecting the resistance heating evaporation source with the surface to be vapor-deposited.

The combination of DeLozanne, Higuchi and Yanai does not teach or suggest these features of claim 5. The above features of claim 5 allow the accelerated electron beam emitted from the electron beam source to perform two functions. Namely, this configuration of claim 5 allows the accelerated electron beam (1) to evaporate the first thin film material (contained in the electron beam evaporation source) by heating and (2) intersect the vapor stream of the second thin film material released by the resistance heating evaporation source in order to ionize the vapor streams of the first thin film material and the second thin film material.

While DeLozanne and Yanai both teach an electron beam source that emits an accelerated electron beam that evaporates a thin film material by heating, neither reference contemplates both evaporating a first thin film material by heating and ionizing a second thin film material by sending the accelerated electron beam along a path that intersects a vapor stream of the second thin film material.

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Higuchi, on the other hand, merely teaches that thermionic beams 41, 42 and 43 are used to ionize vapor streams (see column 3, lines 37-45 and column 4, lines 56-68 of Higuchi) and does not contemplate evaporating a thin film material by heating. In contrast, Higuchi teaches using heating crucibles 11, 12 and 13 (and not electron beam sources) for heating and spouting evaporated metallic elements 1, 2 and 3 (see column 3, lines 23-36 of Higuchi).

Thus, nowhere do any of the cited references contemplate an electron beam source that is configured to emit an electron beam source that (1) evaporates the first thin film material (contained in the electron beam evaporation source) by heating and (2) intersects the vapor stream of the second thin film material released by the resistance heating evaporation source in order to ionize the vapor streams of the first thin film material and the second thin film material, as required by the electron beam source of claim 5. Accordingly, it would not be obvious to one skilled in the art to look at the cited references as a whole and contemplate the above noted features of claim 5.

Also, as discussed in the November 18, 2009 Response, an electron beam used for ionizing a thin film material does not need to be accelerated. However, in order for an electron beam to evaporate a thin film by heating, the electron beam must be accelerated (see highlighted portions of page 3 of Handbook of Thin Film Process Technology, Glocker, David A., Shah, S Ismat, 1999, submitted herewith). As discussed above, Higuchi merely provides the thermionic beams 41, 42 and 43 for ionizing vapor streams. As Higuchi does not contemplate evaporating a thin film material by heating or provide any other reason for emitting an accelerated electron beam, one skilled in the art would not interpret Higuchi as teaching an electron beam source that emits an accelerated electron beam.

The rejection asserts that "Since the electron beams of Higuchi are being radiated from the source toward the vapor streams, they would have to be accelerated toward the vapor streams, thus making the electron beam sources of Higuchi accelerated electron beam sources". However this is incorrect.

Applicants note that electron beam sources are not like vehicles, in that vehicles at rest must be accelerated from zero velocity in order to move. That is, electron beams are not stored in a resting position in an electron beam source waiting to be accelerated in

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order to travel out from the electron beam source. In contrast, electron beams are generally emitted from an electron beam source at a constant velocity and are only accelerated by electron beam sources that provide a voltage for creating an electric or magnetic field in the electron beam source in order to accelerate the electron beam. Higuchi does not use such a system.

Nowhere does Higuchi teach or suggest that its thermionic beam generators 41, 42 and 43 provide a voltage for creating an electric or magnetic field in order to accelerate the thermionic beam. Thus, one skilled in the art would not contemplate that the thermionic beam generators 41, 42, 43 of Higuchi emit an accelerated electron beam, as required by the electron beam source of claim 5.

For at least these reasons claim 5 is not suggested by the combination of DeLozanne, Higuchi and Yanai and should be allowed. Claims 7 and 9 depend from claim 5 and should be allowed for at least the same reasons.

Claim 8 is rejected as being unpatentable over DeLozanne in view of Higuchi and further in view of Suzuki (US Patent No. 4,622,919). This rejection is traversed. Claim 8 depends from claim 5 and should be allowed for at least the same reasons described above. Applicants do not concede the correctness of this rejection.

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Conclusion:

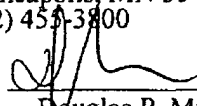
Applicants respectfully assert that the pending claims are in condition for allowance. If a telephone conference would be helpful in resolving any issues concerning this communication, please contact Applicants' primary attorney-of record, Douglas P. Mueller (Reg. No. 30,300), at (612) 455-3804.



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## A1.2 Electron Beam Evaporation

*E B Graper*

### A1.2.0 INTRODUCTION

The electron beam heated evaporation source is a thermal evaporator as is a resistance heated source. Electron beam heated sources differ from resistance heated sources in two ways: the heating energy is supplied to the top of the evaporant by the kinetic energy of a high current electron beam, and the evaporant is contained in a water cooled cavity or hearth. Heating by electron beam allows attainment of temperatures limited only by radiation and conduction to the hearth. Evaporants contained in a water cooled hearth do not significantly react with the hearth, thus providing a nearly universal evaporant container.

The bent beam evaporation source as is now used in thin film fabrication for electronics, optics and research was first envisioned by L. Holland [1] and the modern 270° gun was developed in the early 1960s by Hugh Smith and Charles Hanks of Temescal Corporation [2]. In the late 1960s and 1970s electron beam guns were principally used for the deposition of aluminum metallization on semiconductor devices. Today these sources have been largely supplanted for semiconductors because of the switch to alloy metallization coupled with the development of high rate, easily automated planar magnetron sputtering sources. Magnetron sources are much better suited for alloy deposition and more stable in automated production applications. The use of lift-off metallization technology for gallium arsenide and other high performance devices is causing a resurgence of electron beam deposited semiconductor metallization. The development of the new generation of hard multilayer optical coatings has made electron beam evaporation the technology of choice in optics.

### A1.2.1 EVAPORATION KINETICS

The electron beam heated evaporation source, as used for high technology thin film fabrication (figure A1.2.1) has 3 basic sections: the electron gun, the beam deflection magnetic lens, and the evaporant-containing hearth. The beam is formed in the gun, passes through the magnetic lens and is focused upon the evaporant. The specifics of the design of an electron beam source will be treated in section A1.2.3.

Application of an electron beam heated source is governed by three complex relationships.

- (1) The energy balance of the evaporant charge and the requirement for stable dissipation of the beam energy.
- (2) The complex distribution of the evaporant vapour flux from the evaporant surface caused by the pressure within this vapor and the resultant evaporant surface geometry.
- (3) The ionizing effect of the electron beam, as it passes through the evaporant vapour cloud, impacts the melt surface and is partially reflected from that surface.

For useful evaporation to occur, the evaporant surface must reach a temperature such that the surface vapour pressure is greater than  $10^{-1}$  Torr. At very high evaporation rates, this pressure may reach 10 Torr. The electron beam is conventionally accelerated to 10 kV at a current of up to 1.5 A. This beam impacts an area of 1/4–1 cm<sup>2</sup> with an energy of up to 60 kW/cm<sup>2</sup>. Stable evaporation requires that a thermal equilibrium exist in the evaporant and the energy dissipation must be stable. Energy is dissipated from the evaporant principally by conduction to the hearth, then by evaporant phase change heat and least by thermal radiation. A portion of the beam, often containing considerable energy, is also reflected

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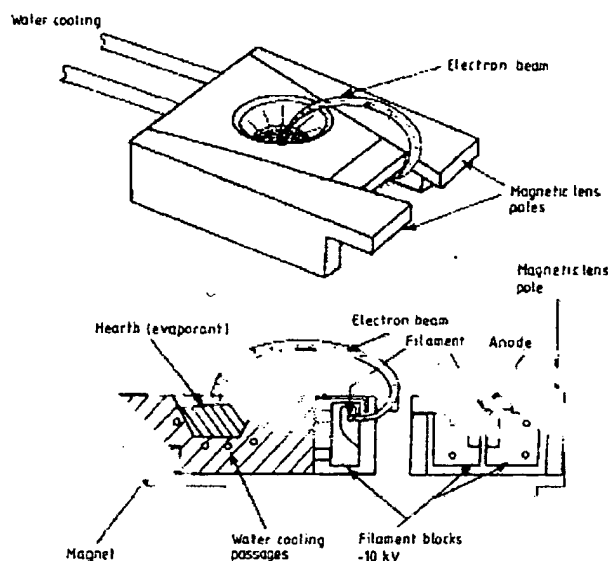


Figure A1.2.1. Electron beam heated evaporation source.

from the evaporant surface [3]. A 10 kV electron beam upon impact with a surface gives up its energy essentially at the surface (within a small fraction of a millimetre). For an ideal evaporant (Ag, Al, Au, Cu), as the temperature is increased, evaporation will begin at a vapour pressure of about  $10^{-4}$  Torr, increasing in rate with increasing energy input until the evaporant pressure over the beam impact point reaches the viscous flow range (about  $10^{-1}$  Torr), and the mean free path is reduced to a fraction of a millimetre. At this point added beam energy is principally absorbed by the evaporant cloud, limiting the further increase in evaporation rate. Because the temperature change in degrees K is relatively small from the point of first evaporation to instability, (for aluminum 1245 K at  $10^{-4}$  Torr to 1640 K at  $10^{-1}$  Torr), heat losses by conduction to the hearth and radiation do not increase rapidly. As a result, the evaporation rate (figure A1.2.2) increases exponentially with increasing power until limited by the vapour density over the evaporant surface. The evaporation rate can be further increased at constant or increased power only by increasing the electron beam impact area, thereby reducing the power density.

Understanding the evaporant distribution of electron beam heated sources is necessary for the production of films of uniform thickness. The flux of evaporant has been described in section A1.0.4, equation (A1.0.14). The rate dependent coefficients are given in section A1.0.4, figure A1.0.5.

Before evaporation begins, an operating electron beam source emits a small current of energetic electrons toward the substrates most likely reflected from the evaporant surface. In addition the electron beam source emits a modest flux of soft x-rays. These emissions from the source, although small, can damage sensitive devices. Because similar or more intense ion and electron bombardment emanates from sputtering sources the most radiation sensitive devices must be coated using resistance heated sources. As the electron beam passes through the evaporant cloud, impacts the evaporant and is absorbed or reflected, several processes occur. Measurements of the ion current at 25 cm above an operating electron beam evaporation source [4] show that the evaporant is partially ionized by the electron beam. For aluminum the ionization is only 1%, but for silver and copper it may be much higher. The path of these ions in the evaporant during their first 1 to 2 cm of travel is influenced by the magnetic lens and the 10 kV beam accelerating field of the evaporation source. Some ions follow curved trajectories, impacting the high voltage feedthroughs and wires, sputtering material and causing arcs. The material sputtered from the feedthroughs is significant

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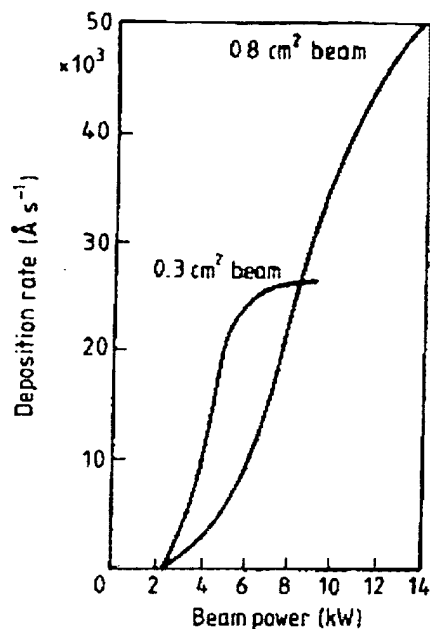


Figure A1.2.2. The aluminum deposition rate (at 25 cm) for a focused 0.03 cm<sup>2</sup> and a diffuse 0.8 cm<sup>2</sup> beam.

enough to destroy them over a period of years and to slightly contaminate the films. These feedthroughs and wires must be protected by a grounded metal shield.

#### A1.2.2. EVAPORATION SOURCES

The electron beam heated source is made up of a power supply and evaporation source. Great care must be taken to assure that their design is sufficiently durable to survive continual arcing and attack by hot molten metal.

The component parts of electron beam heated evaporation sources have, for many years, been made by a number of small suppliers. As a result, the gun/power supply maximum operating specifications have been informally standardized as follows:

~~With Supplies 4 to 10 kV DC~~

Electron beam current: 0 to 1.5 A DC

Filament current: 0 to 40 A AC

Filament voltage: 0 to 6 V AC

Position magnet voltage: -2 to +2 V DC

Position magnet current: 0 to 3 A DC

Emission control voltage: 0 to -10 V DC

The electron beam heated source power supply consists of three interconnected but independent power supplies, high voltage, filament and magnet, as shown in figure A1.2.3. Two designs are used to protect the high voltage supply from arc damage: a regulating pass tube in the ground leg or, less commonly, a switch on the power source or in the ground leg. Most large power supplies are pass tube regulated, using a high dissipation triode. On sensing an arc as an increase in current or a voltage drop across the tube, these pass tubes are shut off sufficiently to limit the current and, if necessary, reduce it to extinguish the arc. These very expensive tubes are durable and capable of actively regulating the high voltage output.

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Switching protected supplies are compact and relatively simple. In all power supplies the high voltage is generated in a vacuum potted 3 phase transformer having a 10–12 kV output and rectified with a full wave bridge using low cost 20 kV avalanche diodes.

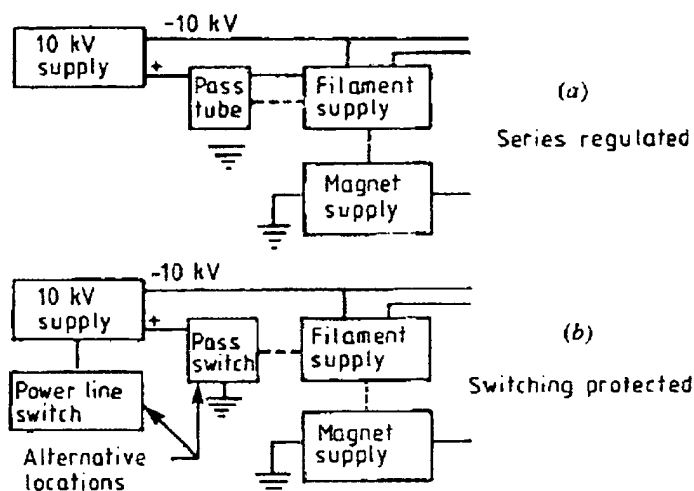


Figure A1.2.3. Electron beam source (a) series regulated and (b) switching protected power supplies.

The filament and magnet power supplies are independent of the high voltage power supply and are unusual only in their exceptional immunity to electromagnetic noise. The SCR filament supply is normally controlled by the 0 to 9 V power control voltage with the output to the gun coupled through a transformer with 20 kV of isolation. There are two feedback paths from the filament supply to the pass tube/switch, one for beam current regulation (if used) and a second to 'fold back' the filament power during arcs to assist arc recovery. The magnet power supply is of basic current regulated design with adjustable current limits, putting out  $\pm 2$  amps and crossing through zero smoothly. This supply is often interlocked with the filament to minimize the chance of the beam being directed outside the hearth.

The high voltage used in electron beam sources and power supplies is exceptionally dangerous. All components must be located with grounded safety shields and a thorough safety interlock system on the cabinet, feedthroughs and vacuum chamber must be used. In addition, the water cooling is critical to operation of both the power supply and sources, requiring water flow metering interlocks and a low flow alarm. Operating an electron beam gun for less than one minute at only 5 kW output without water cooling will usually destroy it.

Electron beam heated evaporation sources are an integrated assembly (figure A1.2.2) of electron gun, magnetic lens and water cooled, evaporant containing hearth. The electron gun, the least integrated module of the evaporation source, consists of a pair of filament holding blocks and a coil filament (of 0.8–1 mm diameter tungsten wire) mounted on ceramic insulators and positioned about 2 mm from a grounded anode. The filament cavity (cathode) and anode configuration are designed empirically for ease of beam extraction and mechanical stability at high temperature. To minimize arc formation in the ion rich vacuum surrounding the cathode it must be carefully fabricated, and molybdenum is usually the material of choice. Because the filament blocks operate at up to 500°C, the alumina mounting insulators must be of high purity, carefully shielded and heatsunk on the grounded end.



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The magnetic lens and the evaporant containing hearth are usually a totally integrated assembly, though for clarity they will be treated separately in this discussion. A magnetic field of a few hundred gauss is required to bend the electron beam from the electron gun through  $270^\circ$  and focus it upon the evaporant. The magnetic circuit consists of a pair of soft iron side plates with adjustable beam formers and two magnets, an electromagnet and a permanent magnet, in parallel. The field of the permanent magnet is chosen such that the beam is positioned within the hearth, and the parallel electromagnet is used to increase and decrease the main magnetic field for longitudinal beam position control. Like the cathode cavity, the magnetic lens is empirically designed to focus the beam to about 0.5 cm diameter at a nearly vertical angle of incidence upon the evaporant charge. The principal focusing elements of the magnetic lens are the beam formers [5] which create an adjustable sharp field concentration and gradient. This focus adjustability is of critical importance, as discussed in section 1.2.4, for the evaporation of dielectrics. For lateral beam positioning, a small secondary magnetic circuit is often included, particularly in large hearth (over  $20 \text{ cm}^3$ ) sources. An electromagnet with one pole near the electron gun and the other loosely coupled to the side plates performs this function. The required field is very low, and no detailed lens design is necessary. All the permanent and electromagnets in a evaporation source must be embedded within the water-cooled hearth or incorporate water cooling because of the high ambient temperature. Particular care must be taken in winding the electromagnets or they will be damaged by voltage spikes induced into them by high voltage arcs.

The core of an electron beam heated source is the hearth assembly, upon which the electron gun and magnetic lens are mounted. The water-cooled hearth, containing the evaporant, also serves as the mounting base for the entire gun. The gun hearth must make excellent electric contact to the vacuum chamber base because the ground leg of the high voltage flows through it. A good earth ground, that is connected to the power supply and the vacuum chamber by a large (5 cm) ground strap is often required to control RF arcs. As the container for the superheated evaporant, the hearth must, above all, have sufficient water cooling to be inert. It has been shown that for aluminum, a particularly reactive metal, evaporated from a well-designed hearth, there is no detectable hearth-related contamination [6]. The conventional hearth water cooling consists of a series of narrow water passages that require flow of 15 l/min. Flow velocity is the key aspect of the cooling, rather than water temperature, and great care must be taken to insure the actual flow rate is sufficient at all times. Because the water may carry away up to 90% of the beam energy, any interruption or reduction in flow during operation can destroy the hearth.

An important extension of the basic single hearth source is the multihearth source. By fabricating the hearth as a rotary turntable with four or more independent hearth cavities and a water-distributing central axis, a great increase in versatility can be achieved. Most films today are multilayer films, and multihearth sources are commonly used, particularly for optics. These hearths are significantly larger and more complex to design and fabricate than single hearth sources. Of special importance is the O-ring sealed central rotary water joint. Unless this joint is designed with exceptional stiffness and close tolerances, it will tilt or unduly wear the O-rings during rotation, and subsequently leak. For reliable service careful maintenance and lubrication of the hearth O-rings is necessary. The magnetic fields couple easily across a 1 mm gap between the fixed magnetic lens and that part of the lens incorporated into the rotating hearth.

In addition to containing the evaporant, the hearth must contain the electrons reflected from the evaporant charge. Because the energy in the reflected electron beam may contain 30% of the beam energy, capturing these electrons is of significant importance. This is usually done by mounting the permanent magnet on top of the hearth, behind a water-cooled copper shield, in the path of the reflected electrons. This creates a strong magnetic field concentration which bends these electrons into the shield over the magnet, capturing them.

### A1.2.3 EVAPORATION CHARACTERISTICS

Success in depositing films using electron beam heated sources is much more sensitive to application technology than is the case for sputtering. The evaporant quantity required by an electron beam heated source may be as little as  $1 \text{ cm}^3$ , yet the selection and conditioning of the evaporant are of utmost importance for successful film deposition.

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The conditioning of the evaporant consists of a slow increase in power (or rate for subliming materials) over several minutes to one and one half to two times the level desired before opening the shutter to clean and degas the charge. Occasionally, during this process, a melting material will give off such large quantities of gas that the system pressure will rise above the maximum acceptable ( $4 \times 10^{-5}$  Torr for example), and degas very slowly, even at high power. Experience has shown that by repeatedly pulsing the power to the highest practical for a few seconds, then when the pressure reaches the maximum allowable, or the charge becomes unstable, reducing the power by 90% and allowing the system to recover, significantly speeds conditioning. Severe gas evolution may also be a sign the evaporant is decomposing. Each incremental power increase is a testing process in which the evaporant charge must be examined for stability and spitting by using a small mirror mounted within the vacuum chamber. In some cases, extreme patience is required as the conditioning may require over an hour. Conditioning is completed by running the gun at maximum conditioning power, while sweeping the beam back and forth over the gun charge and watching for instability in the charge. Semimelting materials (those melting only at the point of electron beam impact) are particularly difficult to condition, as it is usually impossible to melt the entire charge volume. As semimelting materials are consumed by evaporation, new subsurface material is melted and exposed to the substrates, often with violent instability. This can be minimized by conditioning the charge in several pumpdowns, removing it and turning it 90° after each pumpdown. With subliming materials, the amount of conditioning that occurs is necessarily low, and slow due to low gas mobility in solids.

The deposition rate is strongly influenced by the variable characteristics of the electron beam gun and the evaporability of the material [7]. The influence of the electron beam gun hearth size and beam spot size is seen in table A1.2.1. For melting materials (those which melt before evaporating, Al, Au, Cu), the rate increases with increased power density (decreased spot size) in the melt, to the power dissipation limit. With semimelting materials (those which melt only in the beam, sapphire), and subliming materials (chromium and silicon monoxide), which are not able to absorb the full power of the beam, the power and rate can be increased only by increasing the spot area. For these materials a fixed beam spot is similar to a swept one of equal area. The small spot, swept at 60 Hz, though to the eye a good approximation of a fixed large spot, is still only an approximation and is not fast enough to allow nearly as much power into silicon monoxide as does the fixed spot at 1 cm<sup>2</sup> area.

Table A1.2.1. The effect of electron beam gun parameters on the deposition rate of representative materials. Column headings in cm<sup>2</sup> are spot sizes. Rates in Å/min; power in kW.

Material	State at evaporation	Maximum deposition					
		3.2 cm <sup>3</sup> hearth		30 cm <sup>3</sup> hearth			
		1/4 cm <sup>2</sup>	1 cm <sup>2</sup> <sup>a</sup>	1/4 cm <sup>2</sup>	1 cm <sup>2</sup> <sup>a</sup>	1 cm <sup>2</sup>	2 cm <sup>2</sup> <sup>a</sup>
Aluminum wire	Melts	5000	3000	10 000	5000	5000	2000
		6	6	6	6	6	6
Sapphire chunk	Semimelts <sup>b</sup>	4000	6000	4000	5800	6000	10 000
		2	3	2	3	3	4
Chromium Hot	Sublimes	800	3300	800	3000	2800	12 000
Pressed		0.7	1	0.6	1	0.9	1.3
Silicon Monoxide Hot	Sublimes		3000		3100	6000	41 000
Pressed			0.3		0.3	0.4	0.6

<sup>a</sup> Beam spot swept at 60 Hz.

<sup>b</sup> Melts at beam impact point only.

<sup>c</sup> Rate very low or zero due to material spitting or exploding from the charge regardless of power.

The melting materials deposit at higher rates from the large hearth due to the longer thermal path to the water cooling. Some subliming materials also exhibit increased rates in the large hearth, due perhaps to

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the larger charge permitting heating a greater volume to evaporation temperature. With all dielectrics, the beam power must be increased slowly (30 seconds to 1 minute) to red heat, allowing the electrical conductivity to increase to a point where the surface charge on the material can dissipate to ground.

The charge form affects strongly the evaporability of the materials (see table A1.2.2). Materials that are good thermal conductors and melt well below evaporation temperature, e.g. zinc, can be evaporated from any form of raw material. All other materials are difficult or impossible to evaporate from anything but chunks or hot-pressed pieces. Most materials, when heated as powders or granules, form a localized hot spot of very small volume at too low a power to permit conditioning or significant evaporation. The large surface area of powders and granules also permits the absorption of large amounts of water vapour and other contaminants which are released upon heating, causing localized exploding from the gun.

Table A1.2.2 The effect of charge form on the deposition rate of representative materials.

Material	State at evaporation	Charge form	Maximum power (kW)	Maximum <sup>a</sup> rate (Å/min)
Chromium	Sublimes	Granular	<sup>c</sup>	<sup>c</sup>
		Hot pressed	1.3	12 000
Silicon monoxide	Sublimes	Granular	<sup>c</sup>	<sup>c</sup>
		Chunks	<sup>c</sup>	<sup>c</sup>
		Hot pressed	0.6	41 000
Silicon	Semimelts	Chunks	<sup>c</sup>	<sup>c</sup>
		Lumps <sup>b</sup>	6.0	7000
Zinc	Melts	Powder	1.5	39 000
		Chunks	1.5	39 000
		Wire	1.5	39 000

<sup>a</sup> At optimized conditions for each material, from evaluations.

<sup>b</sup> Entire charge was on solid piece.

<sup>c</sup> Rate very low or zero due to spitting or exploding from the charge regardless of power.

The detailed evaporation characteristics for a wide range of materials are reported in table A1.2.3 [8]. In this table, the evaporation rate reported is not the maximum but a practical rate obtainable without significant charge instability. The state at evaporation is tabulated at the indicated deposition rate. At significantly different rates the state may be different. For example, titanium sublimes at very low rates and magnesium melts at extremely high rates.

'Stability' is perhaps an excessively subjective term to use, but it is also the one which best describes the most desirable behaviour. Stability, as used in table A1.2.3, is graded from excellent for materials that, once conditioned, can be evaporated with virtually no risk of spitting or melt explosion, to poor for materials that, though evaporable, require intensive attention and may explode or avalanche at any moment. A material avalanches when a slight increase in rate decreases the impedance of the electron beam gun, resulting in an uncommanded increase in power (beam current) followed by a further increase in rate, perhaps continuing to the point of emptying the hearth in moments at a rate of thousands of angstroms per second.

The 'visual clues' in table A1.2.3 are what will be seen in the mirror during evaporation. Those materials listed as 'none' evaporate or sublime below the temperature of incandescence and do not fluoresce. The listed hazards are only the most serious and acute encountered during film deposition. All materials used in thin films are hazardous in some context and all must be investigated for hazard before use. Particular care must be exercised during equipment clean-up to minimize exposure to toxic materials, minimize the risk of fire, and to assure safe disposal of waste.

The following are four widely applicable guidelines for successful deposition of thin films from an electron beam heated evaporation source.

- (1) Select a charge form with the largest possible volume to area ratio. Avoid trying to evaporate powdered

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or granular materials.

- (2) Use the largest hearth volume consistent with available evaporant charge and the desired film.
- (3) Use the largest beam spot area possible but still attain the required deposition rate.
- (4) Increase the spot size if increasing the beam power causes instability or film pinholing.

### REFERENCES

- [1] 1951 British patent 754 210
- [2] 1969 US patents 3230 110
- [3] Yamagishi K 1965 *Proc. 1st Int. Conf. on Electron Ion Beam Science* ed R Bakish (New York: Wiley) pp 245-63
- [4] Graper E B 1970 Charged particle flux generated by an electron-beam deposition source *J. Vac. Sci. Technol.* 7 (1) 282-5
- [5] Hanks C W 1969 US patent 3483 417
- [6] Graper E B 1972 Deposition of aluminum from an electron beam source *J. Vac. Sci. Technol.* 9 (1) 33-6
- [7] Graper E B 1971 Evaporation characteristics of material from an electron-beam gun *J. Vac. Sci. Technol.* 8 (1) 333-7
- [8] Graper E B 1987 Evaporation characteristics of material from an electron-beam gun II *J. Vac. Sci. Technol. A* 5 (4) 2718-23

## Electron Beam Evaporation

TABLE A1.2.3 The Evaporation Characteristics of Materials from an Electron Beam Heater Source [8]

Evaporant	Symbol	Charge Form	Conditioning Time (min)	State at Deposition (A/sec @ 10 <sup>3</sup> )	Stability	Similar To	Film Appearance	Visual Clues	Hazards	Notes
Aluminum	Al	Wet/Lump	1	Melts	20 @ .5	Excellent	Au	White	Red	...
Aluminum Oxide	Al <sub>2</sub> O <sub>3</sub>	Supplive	3	Smelted	10 @ .5	Good	SiO <sub>2</sub>	Clear	Very Bright	...
Antimony	Sb	Lump	2	Melts	50 @ .5	Avalanches	...	Gray	None	Start slowly; sapphire will explode.
Antimony Trioxide	Sb <sub>2</sub> O <sub>3</sub>	Powder	2	Sublimes	10 @ .2	Good	...	Clear	None	Melt viscous. Use Mo liner.
Arsenic	As	Lump	None	Sublimes	Unstable 100 @ .1	Avalanches	Pb, Se	Silver/Gray	None	...
Barium	Ba	Lump	1	Melts	20 @ .1	Avalanches	Pb, Se	Silver	Fluorescent Blue	Condenses on H <sub>2</sub> trap, contaminates equipment.
Barium Chloride	BaCl <sub>2</sub>	Mo to Mo <sup>6</sup>	1	Melts	30 @ .4	Good	...	Clear	Fluorescent Blue	Getters
Barium Fluoride	BaF <sub>2</sub>	Powder	3	Sublimes	50 @ .1	Good	...	Clear	Fluorescent Blue	...
Beryllium	Be	VM	2	Melts	100 @ 1.5	Excellent	Al	Gray	Bright	Extremely toxic
Beryllium Oxide	BeO	WPC	2	Melts	40 @ 1	Excellent	...	Clear	Transparent	Difficult to degas.
Bismuth	Bi	Lump	2	Melts	100 @ .5	Excellent	...	Gray	Pale Red	...
Boron	B	Lump	13 hr	Melts	10 @ 1.5	Explosive	Unique	Black	Very Bright	Start @ 1 Ang/sec; do not exceed 10 Ang/sec. Watch for arcing on cooling; remove and replace each run.
Boron Carbide	B <sub>4</sub> C	Lump	3	Smelted	35 @ 1	Smooth	Cr	Black	Very Bright	...
Cadmium	Cd	Shot	1	Melts	30 @ .3	Avalanches	...	White	None	Destroys mirror @ .5 micron. Use Mo liner.

## Thermal Evaporation

Evaporant	Symbol	Charge Form	Conditioning Time (hrs)	State at Deposition (M/mic @ 10")	Stability	Similar To	File Appearance	Visual Clues	Hazards	Notes
Cadmium Sulfide	CdS	Lump	3	Sublimes 10 @ .25	Smooth	...	Yellow	Fluoresces Purple	Toxic	
Calcium	Ca	Shot	1	Sublimes 20 @ .2	Poor	Mg	Gray	None	Flammable	Spits occasionally throughout evaporation.
Calcium Fluoride	CaF <sub>2</sub>	1cm Pieces	3-5	Sublimes <sup>d</sup> 30 @ .05	Good	...	Clear	Bright Red	...	Heat to red in air before using.
Carbon	C	See Rod	1	Sublimes 30 @ 1	Poor	Cr	Black	Very Bright	Eye Protection	Covers entire jar and tooling. Required
Chromium	Cr	Shot	.5	Sublimes 15 @ .3	Good	...	Silver	Bright	...	May split, melt at hotbase points.
Cobalt	Co	Wt	3	Melts 20 @ 2	Fair	Ti	Gray Silver	Very Bright	...	Clean hearth after every run.
Copper	Cu	See Rod	1	Melts 50 @ .2	Good	Ag	Copper	Bright	...	Gatters. Use oxygen-free Cu. Use Mo liner.
Dysprosium	Dy	Lump	2	Sublimes <sup>d</sup> 30 @ .3	Excellent	Sc	Black	Bright	Flammable	Gatters, burns in air.
Europtium	Eu	Lump	3	Sublimes in oxide 15 @ .4	Poor	...	Silver/Black/Gray	Oxide breaks w/blue puffs	Flammable	Eu packed in heavy oil with thick oxide.
Gadolinium	Gd	Lump	1	Melts 30 @ 1.5	Excellent	Ti	...	Bright	Flammable	Gatters, burns readily in air.
Germanium	Ge	1cm Pieces	3	Melts 25 @ 3	Good	Si	Black	Bright	...	
Gold	Au	Any	.5	Melts 30 @ 6	High	Cu, Al	Gold	Bright	...	Avoid C liners. Will oscillate.
Inconel Ni/Cr/Fe	Wt	...	3	Melts 50 @ 3	Good	Ni	Gray	Bright	...	Very low rates best, will split.
Iridium	Ir	Any	1	Melts 100 @ .1	High	Sn, Pb	Metallic White	Fluoresces violet before melting	...	Use Mo liner. Filings may melt.
Iridium	Ir	See Rod	3	Melts 10 @ 3	Good	Pt	White	Very Bright	...	
Iron	Fe	Wt	2	Melts 50 @ 2.5	Low	Ni	Gray	Bright	...	Shots gun magnets.
Lanthanum	La	Lump	3	Melts 100 @ 3.5	Good	Ti	White	Bright	Flammable	Burns in air.
Lanthanum Boride	LaB <sub>6</sub>	IP	5	Sublimes <sup>d</sup> 100 @ 1.5	Low	ZrO <sub>2</sub>	Black	Very Bright	Toxic	Power very unstable. Gatters.

## Electron Beam Evaporation

Evaporant	Symbol	Charge Form	Conditioning Time (min)	State at Deposition (A/sec @ 1W)	Stability	Similar To	File Appearance	Visual Clues	Hazards	Notes
Lead	Pb	Any	2	Melts	30 @ .1	High	Sn, Zn	Shiny Black	Toxic	Use Mo liner. Film oxidize quickly.
Lithium	Li	Shot	1	Melts	100 @ .05	High	Zn	Gray Matte	Flammable	Use Mo liner. Vent to Ar, not N <sub>2</sub> .
Lithium Fluoride	LiF	Mo	3	Melts, Viscous	10 @ .15	Poor	BaCl	Clear	..	Use Mo liner. Spits easily; bubbles at high rate
Magnesium	Mg	1oz Rod	None	Sublimes	100 @ .04	Poor	..	Fluoresces Green	Flammable	Do not use turnings or ribbon.
Magnesium Fluoride	MgF <sub>2</sub>	VM Lures	1	Semimelts <sup>d</sup>	30 @ .01	Good	..	Fluoresces White	..	..
Manganese	Mn	1-5oz Pieces	3	Semimelts <sup>d</sup>	40 @ .4	Fair	Mo	Black	Flammable	Vent to H <sub>2</sub> @ room temperature.
Molybdenum	Mo	VM	5	Melts	40 @ .4	Fair	W	Gray	Very Bright	..
Niobium	Nb	1oz Pieces	5	Melts	20 @ 1.4	Good	Co	White	Bright	Flammable Goggles
Nickel	Ni	VM	5	Melts	25 @ 2	Poor, Spits	Ti	Gray/White	Bright	..
Niobium	Nb	Any	3	Melts	20 @ 5	Good	Ti	Gray/White	Very Bright	Spits most severely of all common materials.
Palladium	Pd	4oz Rod	5	Semimelts <sup>d</sup>	100 @ 2	Good	W	White	Bright	Eye Protection Very hot. Will spit late in run.
Platinum	Pt	3oz Rod	3	Melts	20 @ 4	Good	Ti, Au	White	Bright	..
Potassium Chloride	KCl	VM in Mo	1	Semimelts <sup>d</sup>	30 @ .05	Good	BaCl	Clear	Red	Eye Protection Required
Potassium Fluoride	KF	VM in Mo	1	Sublimes	40 @ .05	Poor	SiO	Clear	None	..
Praseodymium	Pr	1-5oz Pieces	5	Melts	25 @ 1.5	Good	..	White	Bright	Deliquescent. Beam tunnels thru charge easily.
Rhenium	Re	3oz Rod	15	Melts	3 @ 3	Poor	KI	Gray/White	Bright	Flammable Oxidizes easily; films react to brown. Gassy at melt.
Scandium	Sc	5oz Pieces	2	Semimelts <sup>d</sup>	50 @ .5	Good	Ti	White	Bright	Eye Protection Spits very badly; VM material not available Required
Selenium	Se	Shot	None	Semimelts <sup>d</sup>	Unlabeled	Excellent	..	Black	None	Flammable Goggles
Silicon	Si	1-5oz Chips	10	Melts	15 @ .15	Poor/Explosive	Os	Black	Bright	Very Toxic High rates consume chamber. Clean hearth after every run.

## Thermal Evaporation

Evaporant	Symbol	Charge Form	Conditioning		Stability	Similar To	Film Appearance	Visual Clues	Hazards	Notes
			Time (min)	State at Deposition (A/mc @ 1at)						
Silicon Dioxide	SiO <sub>2</sub>	1-5m Chips	2	Sublimates <sup>d</sup> 20 @ .7	Good	Cr	Clear	Bright	...	Commercial quality not spotty
Silicon Monoxide	SiO	5mm Pellets	1	Sublimes 20 @ .1	Poor	Cr	Clear	Red	...	
Silver	Ag	Shot	1	Melts 100 @ .5	Good	Cu	Silver	Silvery	...	Use Mo liner.
Sodium Chloride	NaCl	4m in Mo	3	Sublimates <sup>d</sup> 50 @ .15	Good	BaCl	Clear	Fluorescent	...	Films stable, use U.S.P. sodium chlor ide.
Sodium Fluoride	NaF	4m in Mo	3	Sublimates <sup>d</sup> 40 @ .2	Good	BaCl	Clear	Fluorescent Green	...	Toxic
Strontium	Sr	1cm Rod	1	Melts 50 @ .1	Poor	Mg	White	Fluorescent Blue	...	Toxic
Sulphur	S	4m in Mo	3	Melts 100 @ .02	Poor	Zn	Gray/ Yellow	None	...	Melts under oxide, which collapses. Causes exceptional equipment contamination. Minimize vacuum exposure as film evaporates.
Tantalum	Ta	5mm Rod	3	Sublimates <sup>d</sup> 100 @ 6	Good	Mo	Gray	Very Bright	...	Eye Protection Extremely hot
Tin	Sn	Shot	2	Melts 10 @ 2	Excellent	Cu	Gray	Red, Beam Flickers	...	Required
Titanium	Ti	4m	3	Melts 20 @ 1.5	Good	Co	White	Bright	...	Use Mo liner. Fog mirror.
Titanium Dioxide	TiO <sub>2</sub>	HP	5	Melts 10 @ 1	Good	..	Clear	Bright	...	Getters
Tungsten	W	4cm Rod	5	Melts 20 @ 5.5	Good	Co	Black	Very Bright	...	Films absorbing @ high rates, O <sub>2</sub> atmosphere required
Tungsten Trioxide	WO <sub>3</sub>	HP	5	Sublimes 30 @ .1	Good	SiO	Yellow	Red	...	Film Deposits on extremely hot. Required
Vanadium	V	4m	3	Melts 100 @ 4.5	Good	Co	Black	Very Bright	...	Getters
Ytterbium	Yb	Lump	3	Sublimes 20 @ .1	Good	Mg	Gray/ White	...	...	Numerous small flakes of Yb on substrates
Yttrium Oxide	Y <sub>2</sub> O <sub>3</sub>	HP	20	Sublimes 20 @ 2	Good	..	Clear	Red	...	Beam tunnels thru charge easily.
Zinc	Zn	Any	1	Melts 50 @ .25	Good	Pb	Gray	Fluorescent Violet	...	Use Mo liner. Fog mirror.
Zirconium	Zr	4m	2	Melts 20 @ 5	Excellent	Co	White	Very Bright	...	Flammable Will burn on gun in air.
Zirconium Oxide	ZrO <sub>2</sub>	HP	3	Sublimates <sup>d</sup> 20 @ 1	Good	..	Clear	Very Bright	...	Eye Protection Beam tunnels thru charge easily. Required

<sup>a</sup>Mo-Vacuum melted slugs 3-10 mm.  
<sup>b</sup>4m in Mo - Reagent powder air melted in moly liner.  
<sup>c</sup>4m - Hot press pieces 3-10 mm.  
<sup>d</sup>Sublimates - Melts at the point of beam impact only.  
<sup>e</sup>None - Evaporates or sublimes without incidence of fluorescence.